Predictive Model for Tensile Properties of Carbon Fiber-Reinforced Polymers at Various Moisture Contents

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Abstract

A model to estimate the tensile properties of carbon fiber-reinforced polymers (CFRP) based on their moisture contents and molecular structures is formulated and studied. In the proposed model, water molecules absorbed by polymers are considered congregated near the sub-micro-cavities within the polymers. The absorbed water molecules form hydrogen bonds with the polymer molecules and cause the regions around the sub-micro-cavities to have a reduced elastic modulus. The additive group contributions for polymers are used to determine the contributions of hydrophobic and hydrophilic functional groups separately. This enables the effects of water molecules on the mechanical properties of polymers to be quantified and estimated. For CFRP laminae, an additional effect of moisture on the fiber-matrix interface is considered in the calculation of the transverse tensile strength. The tensile properties are calculated using the proposed model agreed well with the experimental values. It is expected that the time and materials required to obtain the mechanical properties of CFRP exposed to humid environments can be reduced with the help of this model.

Keywords

Mechanical Properties; Carbon Fiber-Reinforced Polymer Composites; Epoxy; Moisture

Introduction

Carbon fiber-reinforced polymers (CFRPs) are composites consisting of a polymer matrix and carbon fibers as reinforcement. Epoxy resin is commonly used as the matrix in CFRPs because of its high resistance to corrosion, good mechanical and thermal properties. The combination of strong carbon fibers with light weight polymer matrix results in CFRPs offering very high strength to weight ratio compared to metals. This is also the primary reason why CFRPs are increasingly being used in aerospace as well as offshore structures. However, the mechanical performance of CFRPs is affected by their storage and service environment. In particular, high humidity can lead to water being absorbed into the CFRPs and result in reduced mechanical strength (Ray and Rathore 2015; Shen and Springer 1977a). Therefore, in order to utilize CFRPs for structures exposed to moderate to high humidity, the effects of the moisture content on the mechanical properties of CFRPs need to be understood and be able to estimate.

Many experimental studies have been carried out to measure the mechanical properties of CFRPs with various water content levels. It is found that the fiber-dominated properties of CFRPs do not change significantly with water content because carbon fibers do not absorb water (Browning 1972; Shen and Springer 1977b; Selzer and Friedrich 1997). A compilation of experimental results on the ultimate strengths and elastic moduli of 0°, 45° and 90° CFRP laminates with various water content levels was presented by Shen and Springer (Shen and Springer 1977a; Shen and Springer 1977b). The ultimate tensile strengths of the 90° laminates were found to be significantly reduced (from 40 to 60% reduction) with increasing water content. For the tensile moduli of the 90° laminates, there is a large scatter in the results where some authors found negligible change in the modulus while others found a reduction of up to 50%. Selzer and Friedrich reported similar results for CFRPs with epoxy matrix. However, for CFRP laminates with polyetheretherketone (PEEK) as matrix, the authors found that the effect of water on the transverse tensile strength was insignificant (Selzer and Friedrich 1997).

Studies on the absorption and diffusion of water in polymers can give insight onto how water affects the

mechanical properties of polymers. Water diffusion can be described by the expression shown in Equation (1) (Nogueira et al. 2000):

$$\frac{M_t}{M_s} = kt^\alpha \qquad (1)$$

Where M_t is the mass of water absorbed at time t, M_s is the mass of water absorbed at saturation, k and α are system parameters. For epoxy based resin, water diffuses into the epoxy and epoxy-based composites following Fickian diffusion where $\alpha = 0.5$ (Ray and Rathore 2015; Nogueira et al. 2000; Zafar et al. 2012). Fick's law given by Equation (2) can be used to predict the water content within the laminate:

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \qquad (2)$$

Where C is the moisture content, D is the diffusivity through the laminate thickness. Moisture content, C can be calculated from the weight of the polymer or composite sample using Equation (3) (Zhong and Joshi 2015):

$$C = \frac{W_f - W_i}{W_i} \times 100\ \% \qquad (3)$$

Where W_i is the initial or dry weight and W_f is the final or wet weight. The diffusion of water is affected by void volume fraction, hygrothermal temperature history (Mikols et al. 1982; Choi et al. 2001) and cure temperature (Pethrick et al. 1996). Pethrick et al. used dielectric measurements to study the water-epoxy interactions and found that water exists in two different states in epoxy: water is bound to epoxy molecules via hydrogen bonds and water molecules that form clusters in the epoxy sub-micro-cavities (Pethrick et al. 1996). The water-epoxy interaction leads to a plasticization effect on the epoxy resulting in a decrease in glass transition temperature, T_g (Moy and Karasz 1980). The mechanical properties of neat epoxy are also reduced with the increase of water content (Nogueira et al. 2000; Zafar et al. 2012).

Besides the polymer matrix, the fiber-matrix interface in CFRPs is also affected by water. The damage mechanism at the interface can include debonding, as well as changes in the surface energy and residual stresses (Crossman, Mauri and Warren 1979). Ramirez, Carlsson and Acha studied the fiber-matrix interface using the single fiber fragmentation test (Ramirez, Carlsson and Acha 2008). They found that moisture caused significant reduction in the interface shear strength as well as debonding. This was translated to a large decrease in the transverse tensile strength of the CFRP laminates. Totten studied the tensile strength at the fiber-matrix interface by performing tests on transverse tensile single-fiber specimens (Totten 2015). The fiber sizing was reported to have a large effect on the interface strength; and strong adhesion at the interface can reduce the degradation caused by water.

Experimental studies have provided valuable insights into the degradation of CFRP mechanical properties due to water. However, the process of conditioning CFRP specimens to the required moisture level for experiments can be very time consuming. The conditioning temperature can be increased to accelerate the process, but this can also lead to unreasonably high moisture content in some cases (Crossman, Mauri and Warren 1979).

It is noted that there is no coherent and specific model that can link the absorbed moisture and the reduction in the properties of CFRP and quantify them. In this study, a method to estimate the ultimate tensile strengths of polymer and CFRP laminae based on their moisture content and molecular structures is introduced. A model is formulated and used for CFRP to investigate their transverse tensile strength. The longitudinal strength is generally controlled by fibers and not affected much by moisture.

Effective Additive Group Contributions for Polymers Affected by Moisture

Water absorbed into polymer can be categorized into two groups: free water and bound water (Moy and Karasz 1980; Pethrick et al. 1996; Nogueira et al. 2000). For the current model, bound water is considered to be the main cause of the reduction in mechanical properties of the polymer. This is because the bound water molecules form hydrogen bonds with the polymer molecules resulting in a decrease in the hydrogen bonding between the polymer molecules themselves. The decrease in bonding between the polymer molecules leads to a reduction in the mechanical properties of the polymer. The effect of free water on the mechanical properties of the polymer is considered small as compared with that of bound water.

Various physical properties of polymers can be estimated from the sum of contributions made by the functional groups in the polymer (Van Krevelen and Te Nijenhuis 2009). In this study, the sum of contributions is modified to reflect the effect of moisture. This modified sum is termed by the effective additive group contribution, A_{eff}. For polymers with some water content C, the effective additive group contribution A_{eff} can be calculated from Equation (4):

$$A_{eff} = \sum A(hydrophobic) + g(C) \sum A(hydrophilic) \tag{4}$$

Where A represents the additive properties and g(C) is a function related to the moisture content, C. Here, the functional groups in the polymer are divided into hydrophobic and hydrophilic groups. The contribution from the hydrophilic groups in the polymer is modified by the function g(C) to account for the effect of moisture. The hydrophobic groups are not affected by water so their contributions do not change with moisture content.

In order to calculate the elastic moduli and ultimate tensile strengths, the additive properties required are the Rao-function (U_R), the Hartmann-function (U_H), the molar parachor (P_s) and the amorphous molar volume (V_a). The effective additive properties are given in Equations (5) to (8). The values of the additive properties used in this study are obtained from (Van Krevelen and Te Nijenhuis 2009).

$$U_{R,eff} = \sum U_R(hydrophobic) + g(C) \sum U_R(hydrophilic) \tag{5}$$

$$U_{H,eff} = \sum U_H(hydrophobic) + g(C) \sum U_H(hydrophilic) \tag{6}$$

$$P_{s,eff} = \sum P_s(hydrophobic) + g(C) \sum P_s(hydrophilic) \tag{7}$$

$$V_{a,eff} = \sum V_a(hydrophobic) + g(C) \sum V_a(hydrophilic) \tag{8}$$

In this study, a linear relationship between the moisture content and the effective additive group contribution is assumed. When the polymer reaches saturation moisture content, the hydrogen bonding sites of the polymer molecules are assumed to be completely taken up by water molecules. The function g(C) is then given by Equation (9):

$$g(C = C_i) = \frac{C_s - C_i}{C_s} \tag{9}$$

Where C is the moisture content, C_i is current moisture content and C_s is the moisture content at saturation. The function g(C) decreases from 1 to 0 as moisture content increases from 0% to saturation.

The material properties calculated from additive group contributions are only estimates of the actual properties. This is because many factors such as temperature and pressure during the curing process can affect the material properties. Therefore, the properties of the dry polymer or CFRP lamina are used as the reference in the calculation.

Localized Reduced Modulus of Elasticity

The Rao- and Hartmann-functions can be used to estimate the elastic properties of polymer (Van Krevelen and Te Nijenhuis 2009). The bulk modulus K and shear modulus μ are given by Equations (10) and (11):

$$K = \rho \left(\frac{U_R}{V}\right)^6 \tag{10}$$

$$\mu = \rho \left(\frac{U_H}{V}\right)^6 \tag{11}$$

Where the molar volume V = M/ρ, M is the molar mass, ρ is the density, U_R is the Rao-function and U_H is the Hartmann-function. Using the relationship between the bulk, shear and elastic modulus, the modulus of elasticity E can be calculated following Equation (12)

$$E = 9\rho^7 \left(\frac{U_R}{M}\right)^6 \left[\frac{(U_H/U_R)^6}{3 + (U_H/U_R)^6}\right] \tag{12}$$

Water molecules tend to cluster in the micro- or sub-micro-cavities in the polymer structure (Pethrick et al. 1996). The size of the cavities is in the order of 10^{-7} to 10^{-6} m (Gdoutous 1993). Figure 1 shows an scanning electron microscope (SEM) image of the cross- section of a CFRP laminate after hygrothermal conditioning; and cavities with the size of microns can be observed (figure taken from (Zhong and Joshi 2015)). The clustering of water

molecules results in the polymer molecules near the sub-micro-cavities to have a high chance of interacting with the water molecules. Some of the free water molecules in the sub-micro-cavities interact and form hydrogen bonds with the polymer molecules and become bound water molecules. This results in the formation of a region with reduced modulus of elasticity near the sub-micro-cavities due to bound water molecules (magnified view in Figure 2). The localized reduced modulus of elasticity E_{red} can be calculated from Equation (13):

$$E_{red} = 9\rho^7 \left(\frac{U_{R,eff}}{M}\right)^6 \left[\frac{(U_{H,eff}/U_{R,eff})^6}{3 + (U_{H,eff}/U_{R,eff})^6}\right] \qquad (13)$$

Where the effective contributions for the Rao- and Hartmann-functions ($U_{R,eff}$ and $U_{H,eff}$) are obtained from Equations (5) and (6), respectively.
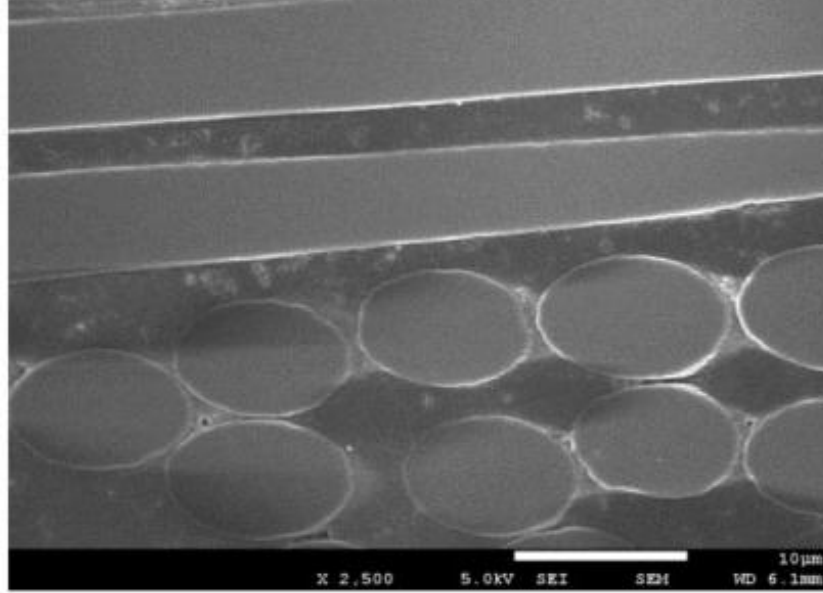

FIG. 1 SEM IMAGE OF THE CROSS SECTION OF A CFRP LAMINATE AFTER HYGROTHERMAL CONDITIONING (Zhong and Joshi 2015). MICRO CAVITIES CAN BE OBSERVED IN THE LAMINATE.

Tensile Properties of Bulk Polymer

A schematic diagram of the water distribution in the bulk polymer is shown in Figure 2. Sub-micro-cavities can be found randomly distributed in the polymer structure. When exposed to high humidity, free water molecules will occupy some of the sub-micro-cavities. The free water molecules can interact with the polymer molecules around the sub-micro-cavities to become bound water molecules. The bound water molecules weaken the polymer structure in the nearby surrounding (region with reduced E in Figure 2).

For the ultimate tensile strength, the region around a sub-micro-cavity in the polymer is considered (magnified view of Figure 2). The cavity can be considered as a sharp crack. The ultimate tensile strength X is taken to be the stress required to cause fracture at the crack. This can be calculated using Griffith's equation shown in Equation (14) (Gdoutos 1993):

$$X = \sqrt{\frac{2\,\gamma_{red}\,E_{red}}{\pi a}} \qquad (14)$$

Where γ_{red} is the reduced surface energy, E_{red} is the reduced elastic modulus given by Equation (13) and a is the cavity size. Two additive properties are required to estimate the surface energy: the molar parachor, P_S and the amorphous molar volume, V_a. The effective molar parachor, $P_{S,eff}$ and effective amorphous molar volume, $V_{a,eff}$ can be calculated from Equations (7) and (8), respectively. The reduced surface energy, γ_{red} is then given by equation (15) (Van Krevelen and Te Nijenhuis 2009):

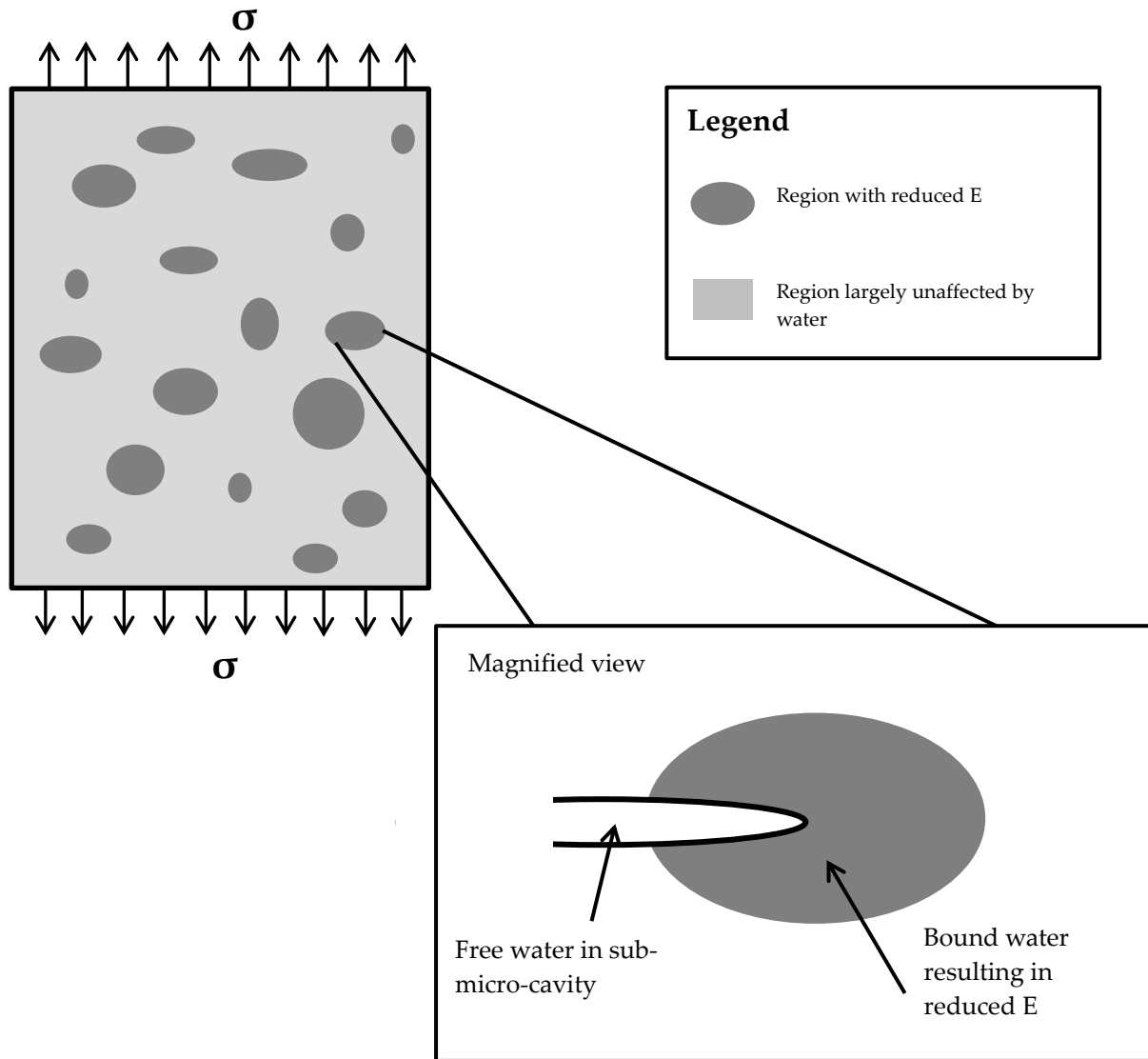$$\gamma_{red} = \left(\frac{P_{S,eff}}{V_{a,eff}}\right)^4 \qquad (15)$$

FIG. 2 SCHEMATIC DIAGRAM OF DISTRIBUTION OF REGION WITH REDUCED E BY WATER IN POLYMER

Seen from experimental results, under the effect of moisture, the degradation of ultimate tensile strength, X is more significant as compared with the degradation of the modulus of elasticity, E (Nogueira et al. 2000; Zhong 2014). This can be explained from the different regions in the model shown in Figure 2. In the regions far away from the sub-micro-cavities occupied by free water, both free and bound water molecules are still present, but they are less concentrated and their effect on the mechanical properties of the polymer is less significant. The region can also include sub-micro-cavities unoccupied by water. Therefore, the regions far away from the sub-micro-cavities occupied by water are considered largely unaffected by water and are assumed to have the same properties as dry polymer. The combination of the different regions results in the degradation of E being less severe than the degradation of X. For the calculation of E for the bulk polymer, the simple rule of mixture is assumed. Using the moisture content calculated from weights to estimate the fraction of region with reduced E in the polymer, the elastic modulus, E at moisture content C_i can be calculated from Equation (16):

$$E_{C=C_i} = E_{dry}\frac{100-C_i}{100} + E_{red}\frac{C_i}{100} \tag{16}$$

Where E_{dry} is the elastic modulus of the dry polymer and E_{red} is the reduced elastic modulus calculated from Equation (13).

Transverse Tensile Strength of CFRP Laminae

The transverse tensile strength X_T of a CFRP lamina can be calculated from the mechanical properties of its

constituents using Equation (17):

$$X_T = \frac{X_m}{S} \tag{17}$$

Where S is the stress concentration factor given by Equation (18):

$$S = \frac{1 - V_f(1 - E_m/E_{ft})}{1 - \sqrt{4V_f/\pi}\,(1 - E_m/E_{ft})} \tag{18}$$

X_m is the tensile strength of the matrix, V_f is the fiber volume fraction, E_m is the matrix elastic modulus and E_{ft} is the fiber transverse elastic modulus (Agarwal, Broutman and Chandrashekhara 2006).

Seen from Equation (17), X_T for CFRP lamina decreases under the effects of moisture since the tensile strength of the polymer matrix X_m reduces. However, the decrease in X_T is larger than the reduction in X_m. This is due to the change in the stress concentration factor. The matrix around the fibers is affected by water and has its elastic modulus reduced (Figure 3). This results in a higher stress concentration. By substituting E_m in Equation (18) with E_{red}, the stress concentration factor can be calculated. The matrix far away from the fibers is modeled to be the same as the bulk polymer under the effects of moisture as discussed in the previous section (Figure 2).
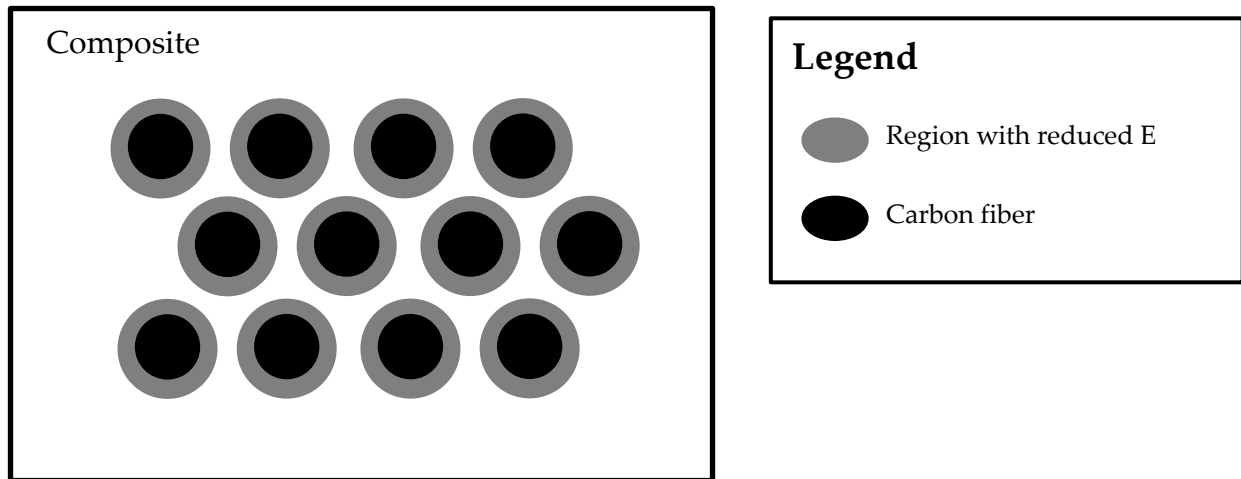

FIG. 3 MOISTURE WEAKENS THE MATRIX AROUND THE FIBERS

Besides this, the fiber-matrix interface bonding is also weakened due to moisture (Selzer and Friedrich 1997; Ramirez, Carlsson and Acha 2008; Totten 2015). For carbon fiber sizing, that is compatible to the matrix, the interface at the matrix side will have properties similar to the matrix. Therefore, for a load applied in the transverse direction, the weakening of the interface bonding can be estimated by Equation (19):

$$F = \begin{cases} 1 & \text{if } C \le \tfrac{1}{2}\,C_s \\ \dfrac{E_{red}}{E_m} & \text{if } C > \tfrac{1}{2}\,C_s \end{cases} \tag{19}$$

Where F is the interface weakening factor, E_m is the dry matrix elastic modulus and E_{red} is the reduced elastic modulus for the matrix given by Equation (13). Mechanical tests have shown that for low moisture content, the reduction in the transverse tensile strength of CFRP laminae is small (Shen and Springer 1977a; Selzer and Fridrich 1997). This can be due to the moisture not being evenly distributed in the laminae. The weakening of the fiber-matrix interface by water occurs at moisture levels above $\tfrac{1}{2}\,C_s$. Therefore, the factor F in Equation (19) is taken as 1 at moisture content below $\tfrac{1}{2}\,C_s$.

The transverse tensile strength of CFRP laminae under the effects of water can then be calculated from Equation (20):

$$X_T(C = C_i) = F\left[\frac{X_{m(C=C_i)}}{S^*}\right] \tag{20}$$

where S* is given by Equation (21):

$$S^* = \frac{1 - V_f(1 - E_{red}/E_{ft})}{1 - \sqrt{4V_f/\pi}\,(1 - E_{red}/E_{ft})} \tag{21}$$

Validation Results and Discussion

The proposed method and the model developed based on that are tested by comparing the calculated properties with the experimental properties. The experimental properties of the dry polymer matrix of CFRP laminae are used as the references for the calculations. The relative error for the property, error (P) is calculated from Equation (22):

$$\text{error}(P) = \frac{|P_{calc} - P_{exp}|}{P_{exp}} \times 100\% \tag{22}$$

Where P_{calc} is the calculated value and P_{exp} is the experimental value.

For the mechanical properties of polymers, two systems were investigated. The first system is an epoxy fabricated from diglycidyl ether bisphenol A (DGEBA) resin and diethylenetriamine (DETA) hardener (Zhong 2014). The second system was cured from a combination of tetraglycidyl-4, 4′-diaminodiphenylmethane (TGDDM) and multifunctional novolac glycidyl ether (EPN) epoxies using a 4,4′-diaminodiphenylsulphone (DDS) hardener (Nogueira et al. 2000; Barral et al. 1998). A comparison of the calculated and experimental mechanical properties is given in Table 1. The saturated moisture contents, C_s are also included.

TABLE 1 ELASTIC MODULUS (E) AND ULTIMATE TENSILE STRENGTH (X) OF POLYMER AT VARIOUS MOISTURE CONTENT

Polymer	Moisture (%)	Experimental		Calculated				
		E (GPa)	X (MPa)	E_{red} (GPa)	E (GPa)	error(E) (%)	X (MPa)	error(X) (%)
DGEBA/DETA Experimental data from (Zhong 2014)	0	3.165	80.17					
	C_s = 3.154	2.609	40.135	0.967	3.096	18.7	36.81	8.30
TGDDM/EPN/DDS	0	1.5	64					
	1.9	1.55	54	1.212	1.495	3.58	57.84	7.11
Experimental data from (Nogueira et al. 2000; Barral et al. 1998)	3.2	1.53	49	1.029	1.482	3.14	53.48	9.15
	4.3	1.49	49	0.883	1.459	2.08	49.71	1.45
	4.7	1.43	44	0.818	1.432	0.13	47.87	8.81
	C_s = 5.22							

For the DGEBA/DETA epoxy, the calculated E is 18.7% larger than the experimental value whereas the calculated X agrees well with the experimental value. The model underestimated the effect of moisture on the elastic modulus of the bulk material. On the other hand, for the TGDDM/EPN/DDS epoxy, both the calculated elastic moduli and ultimate tensile strengths were in good agreement with experimental values with relative errors of below 10%. Seen from moisture content from 0 to 1.9%, there was a small increase in the elastic modulus. Nogueira et al. attributed this increase to a reactivation of the curing process due to high temperature (Nogueira et al. 2000). The effect of temperature is not taken into account in the current model so this effect cannot be predicted.

For the transverse tensile strengths of CFRP laminae, G30-500/5208 and T300/Fiberite 1034 laminae were investigated. The matrix for the G30-500/5208 laminae is based on TGDDM and DGEBA epoxies cured with DDS hardener (Selzer and Friedrich 1997a). For T300/Fiberite 1034, the matrix consists of aromatic diglycidyl esters and aniline derivatives as hardener (Shen and Springer 1977a). The volume fraction (V_f), matrix elastic modulus (E_m) and fiber transverse elastic modulus (E_{ft}) are given in Table 2. The values for E_{ft} shown in Table 2 were calculated using the properties of dry CFRP as reference as the experimental values were not readily available. The experimental and calculated properties of the CFRP laminae are given in Table 3.

TABLE 2 PROPERTIES OF CFRP LAMINAE INVESTIGATED

CFRP	V_f	E_m (GPa)	E_{ft} [a] (GPa)
G30-500/5208 (Selzer and Friedrich, 1997)	0.6	4.24	8.67
T300/Fiberite 1034 (Shen and Springer, 1977a)	0.68	4.1	10.74

[a] E_{ft} values calculated using properties of dry CFRP as reference.

TABLE 3 TRANSVERSE TENSILE STRENGTH (X_T) OF CFRP LAMINAE AT VARIOUS MOISTURE CONTENTS

CFRP	Moisture (%)	Experimental	Calculated				
		X_T (MPa)	E_{red} (GPa)	X_m (MPa)	F	X_T (MPa)	error(X_T) (%)
G30-500/5208	0	50					
Experimental data from (Selzer and Friedrich 1995; Selzer and Friedrich 1997)	0.8	44	3.219	57.11	1	41.31	6.11
	C_s = 1.6	23	2.421	50.03	0.571	18.63	19.00
T300/Fiberite 1034	0	59					
Experimental data from (Shen and Springer 1977a)	0.5	58	3.713	76.62	1	54.49	6.05
	0.75	55	3.532	73.68	1	51.36	6.62
	1	43	3.359	70.80	0.819	39.62	7.86
	C_s = 1.5	33	3.035	65.22	0.740	31.64	4.13

For both composites, the calculated transverse tensile strengths agree well with the experimental values. The highest relative error of 19.00% is for the calculated X_T of G30-500/5208 composite at the saturation moisture content of 1.6%. However, the calculated value only differs from the experimental value by 4.37 MPa and is within the scatter. A comparison of the experimental and calculated transverse tensile strengths of CFRP laminae is shown in Figure 4.

Selzer and Friedrich also studied the mechanical properties of composites with AS4 carbon fibers (AS4) and polyether ether ketone (PEEK) matrix (Selzer and Friedrich 1997). They found that moisture has little effect on the mechanical properties of the AS4/PEEK composite (Figure 4). This can be explained by examining the molecular structure of PEEK. PEEK contains hydrogen acceptors but no hydrogen donors. Therefore, hydrogen bonds cannot be formed between the PEEK molecules and are not important in determining the strength of the polymer. This result is consistent with the proposed model.
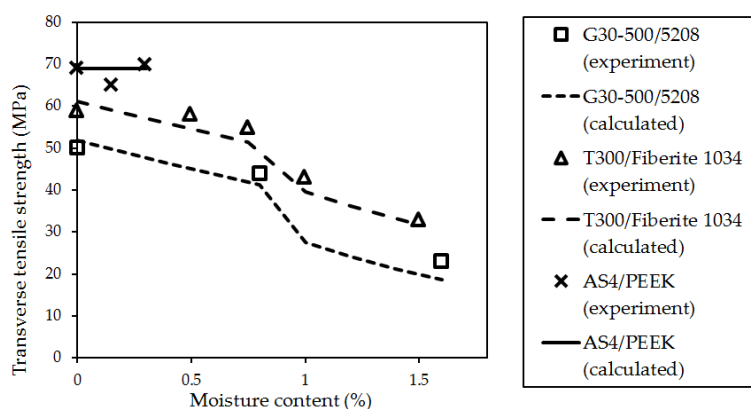

FIG. 4 A COMPARISON OF THE CALCULATED TRANSVERSE TENSILE STRENGTHS OF CFRP LAMINAE WITH EXPERIMENTAL VALUES. EXPERIMENTAL DATA OBTAINED FROM (Selzer and Friedrich 1997; Shen and Springer 1977a).

Conclusions

A method to calculate some of the mechanical properties of polymer and carbon fiber-reinforced polymer composites under the effects of moisture is proposed and a model is established. Water molecules tend to congregate at the sub-micro cavities in the polymer. Based on this finding, the bulk polymer can be modeled as having regions with a reduced tensile modulus. The tensile modulus and ultimate tensile strength can then be calculated from the reduced tensile modulus. For CFRP laminae, the transverse tensile strength is affected by the reduced elastic modulus and strength of the matrix. At the higher moisture levels, the fiber-matrix interface bonding is also weakened due to water and causes further reduction in the transverse tensile strength of the CFRP lamina.

The model has been demonstrated to be able to provide good predictions for carbon fiber-reinforced composites with some of the commonly used polymer matrices. The proposed model enables the tensile strengths of polymer and CFRP laminae at various moisture contents to be predicted based on the molecular structure of the matrix. This will reduce the time and materials required to study the mechanical performance of CFRP structures exposed to a humid environment.

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